PATENT
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IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicant:	Kenji Yamamoto, et al.	Conf.:	5476
Appl. No.:	10/762,462	Art Unit:	1712
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For:	SILICONE COMPOSITION AND A PAPER TREATMENT AGENT COMPRISING THE SAME		

DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Kenji Yamamoto, Annaka, Gunma Prefecture, Japan, hereby declare and state as follows:

I graduated from Kanazawa University, in 1985 to get a master's degree. After graduating from the university, I joined Shin-Etsu Chemical Co.,Ltd. I have been working on silicone in the Silicone-Electronics Materials Research Center. I am a senior researcher in the Development Section No.1.

I am an inventor of the U.S. Application Serial No. 10/762,462, filed on January 23, 2004, entitled "SILICONE COMPOSITION AND A PAPER TREATMENT AGENT COMPRISING THE SAME."

The following experiments were carried out by me to provide evidence of the improved oil repellency of a coating formed from a composition prepared by mixing a silane with water prior to mixing the silane with the other components of the composition.

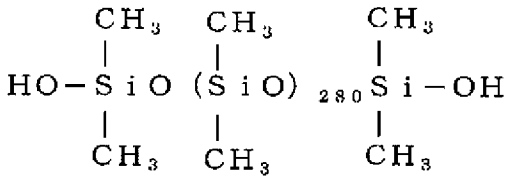
Experimental

Preparation of an aqueous solution of a PVA resin(C)

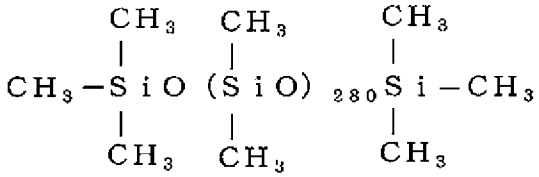
Two hundred parts by weight of a PVA resin having a degree of saponification of 90 mole % and a viscosity in a 4% aqueous solution at 20 °C of 30 mPa·s, and 1800 parts by weight of water were mixed. The mixture was stirred until it became a homogeneous solution to prepare a 10% aqueous solution of the PVA resin.

Preparation of an Emulsion of an organopolysiloxane(A)

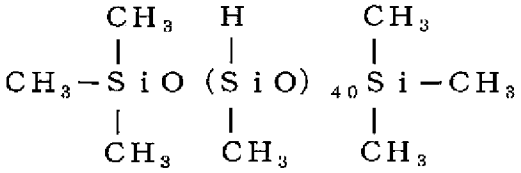
In a 5-liter complex emulsifier provided with an anchor like shape stirrer capable of agitating entire contents of the emulsifier and a rotatable disk having small projections of a tooth like shape directed alternately upperwards and lowerwards, there were placed 95 parts by weight of the organopolysiloxane (A2) of the following formula having a viscosity at 25 °C of 2 Pa·s and a silanol group content of 0.01 mole/100 g,



5 parts by weight of the organopolysiloxane(A1) represented by the following formula having a viscosity at 25 °C of 2 Pa·s,



3 parts by weight of the methylhydrogenpolysiloxane(B)of the following formula having a viscosity of 25 mPa□s and an SiH content of 1.5 moles/100 g,



1 part by weight of polyoxyethylene lauryl ether having an HLB of 13.6 as a surfactant, and 50 parts by weight of the aforesaid 10% aqueous solution of the PVA resin and stirred to make a homogeneous mixture. To the mixture, 10 parts by weight of water were added to cause phase inversion and stirred for additional 30 minutes. Then, 503 parts by weight of water were added to prepare 667 parts of a 15% oil-in-water type silicone emulsion.

Preparation of a mixture of a silane (G) and water(E)

In a 100-ml flask, 30 parts by weight of tetraethoxysilane was placed and heated at a temperature of 50 °C. To the tetraethoxysilane, 30 parts by weight of water was gradually added in 30 minutes while stirring. By stirring for another 300 minutes at room temperature, 60 parts by weight of a transparent aqueous solution was obtained. This solution, hereinafter referred to as "component (G) solution", contained a mixture of hydrolyzed tetraethoxysilane and condensates thereof.

Preparation of Composition (I)

In a mixer, 2000 parts by weight of the aqueous PVA solution,

664 parts by weight of the silicone emulsion, and 60 parts by weight of the component (G) solution and 303 parts by weight of water were mixed thoroughly to obtain Composition (I).

Preparation of Composition (II)

In a mixer, 2000 parts by weight of the aqueous PVA solution, 664 parts by weight of the silicone emulsion, and 30 parts by weight of tetraethoxysilane and 333 parts by weight of water were mixed thoroughly to obtain Composition (II).

Preparation of Paper

Composition (I) was applied with a bar coater to a sheet of commercially available craft paper of 50 g/m^2 in such an amount that a solid content of the composition was 2 g/m^2. The paper was then heated in a dryer at 140 °C for 30 seconds to prepare a sheet of water- and oil-repellent paper I.

Using Composition (II) in place of Composition (I), Paper (II) was prepared in the same manner as described above.

Evaluation

Paper (I) and (II) were evaluated in oil repellency, water repellency, and potassium permanganate consumption according to the following methods.

Oil repellency

Oil repellency of the paper was evaluated with a 3M kit test, TAPPI-RC-338, ex 3M Corp. In the test, a drop of test oils with various kit numbers, containing castor oil, toluene, and heptane in various ratios, are placed on paper and observed whether it penetrates the paper or not. Oil repellency of the paper is represented by the test oil of the maximum kit number which did not penetrate the paper. The greater the kit number, the better the oil repellency.

Water repellency

A contact angle of a drop of water on paper was measured. The larger the contact angle, the better the water repellency.

Results

Results are as shown in the table below.

	Paper (I)	Paper (II)
Oil repellency	13 ≤	9
Water repellency(degree)	100 <	100 <

Discussion

As shown in the above table, Paper (I) showed better oil repellency than Paper (II) and as good water repellency as Paper (II).

A reason for this is considered as follows.

The silane(G) has higher affinity with the organopolysiloxane (A) than the polyvinyl alcohol(C). Accordingly, when the silane (G) is mixed with the organopolysiloxane (A) simultaneously with polyvinyl alcohol(C) and water (E), the silane (G) tends to be present in the organopolysiloxane (A) phase rather than the aqueous phase. On the other hand, when the silane (G) is pre-mixed with water, at least a part of the silane is converted to silanol and/or a condensate thereof which dissolves in water and has higher affinity to the polyvinyl alcohol (C) than the silane. The silanol/or a condensate thereof in the aqueous phase interacts with the polyvinyl alcohol(C) firmly to paper substrate, whereby the oil repellency is improved.

The undersigned declares further that all statement made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statement may jeopardize the validity of above identified application or any patent issuing thereon.

Nov. 2, 2007
Date

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